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drophobic konjak gel
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1. Title of the Invention:
method for production of
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2. Scope of Claim for Patent

1. A method for the production of a foamed hydrophobic konjak gel obtained as by freezing or heating a mixed system of a konjak derivative and a catalyst.

5 2. A method according to claim 1, wherein said konjak derivative produced by the action of an alkali substance on konjak tuber or konjak powder is not enabled by freezing or heating to form a hydrophobic gel.

10 3. A method according to claim 1, wherein said mixed system of a konjak derivative and a catalyst is in an aqueous form or in a solid form.

4. A method according to claim 1, wherein said catalyst is a single species of food fibers or a mixture of two or more species of food fibers.

15 5. A method for the production of a foamed hydrophobic gel substance obtained as by freezing or heating a formed mixed system of a konjak derivative, other substance, and a catalyst.

20 6. A method according to claim 5, wherein said konjak derivative produced by the action of an alkali substance on konjak tuber or konjak powder is not enabled by freezing or heating to form a hydrophobic gel.

25 7. A method according to claim 5, wherein said mixed system of a konjak derivative and a catalyst is in an aqueous form or in a solid form.

8. A method according to claim 5, wherein said catalyst is a single species of food fibers or a mixture of two or more species of food fibers.

30 9. A method according to claim 5, wherein said other substance is a food-grade or nonfood-grade inorganic or organic material.

10. A method for producing konjak by mixing either a

single species or two or more species of food fibers into konjak formed by the action of an alkali substance on konjak tuber or konjak powder.

3. Detailed Description of the Invention

5 A) Field of industrial application

This invention relates to a formed hydrophobic konjak gel and a method for the production of a foamed hydrophobic gel substance and more particularly relates to a method for the production of a hydrophobic konjak gel no longer capable
10 of being reverted to the original liquid or pasty state by adding a single species of food fibers or a mixture of two or more species of food fibers to a konjak derivative possessed of a hydrophilic liquid or pasty state incapable of forming a hydrophobic gel even when subjected to a freezing treatment
15 or a heating treatment at a temperature of not lower than 70°C and formed by the action of an alkaline substance on konjak tuber or konjak powder and subjecting the resultant mixture to a freezing treatment or a heating treatment at a temperature of not lower than 70°C thereby forming a
20 hydrophobic gel, a method for the production of a formed hydrophobic gel substance by causing the aforementioned step of treatment of freezing or heating to be preceded by a procedure comprising such steps as adding and mixing other substance, molding, freezing, and heating, and a method for
25 the production of konjak.

B) Prior Art

The prior art embraces a method of use which is directed toward forming a coagulated mass not easily soluble in water by freezing "reversible konjak" represented in connection
30 with JP-A-56- 209638 titled "food of reversible konjak and addition and method for use thereof and JP-57-219309 titled "method for production of coagulated mass not easily soluble

in water." The present inventors have not known the fact that a konjak derivative incapable of forming a hydrophobic gel by the action of freezing or heating at a temperature of not lower than 70°C is enabled by the addition of a catalyst to
5 form a hydrophobic gel.

Though other related techniques have been introduced to the art as shown below, none of them make a mention of any catalyst.

Invention of JP-A-59-109151, "method for production of
10 coagulated mass not easily soluble in water"

Invention of JP-A-59-204367, "novel food of konjak, food additive, and method for use thereof"

Invention of JP-A-60-19471, "method for use of konjak"

Invention of JP-A-60-19459, "method for use of reaction
15 product of konjak and egg"

Invention of JP-A-58-244822, "method for use of konjak"

Invention of JP-A-59-227267, "method for use of konjak"

Invention of JP-A-60-070776, "method for production of konjak gel substance"

20 C) Problems to be solved by the invention

(1) The conventional konjak derivative of this sort undergoes serious putrefaction when distributed in the proximity of neutrality at a temperature in the range of 0°C - 50°C.

25 It, therefore, required to be distributed frozen.

(2) The konjak derivative mentioned above requires to be endowed with a nature of being enabled to form a hydrophobic gel by freezing or heating, namely the perfect reverse of the nature existing during distribution.

30 (3) The konjak derivative mentioned above ought to be distributed in a solid state (powder, grains, pellets, flakes, etc.).

In short, the conventional konjak derivative formed by the action of an alkaline substance on konjak powder is characterized by forming a hydrophobic gel by freezing or drying. The present invention is aimed at producing a konjak derivative not enabled to form a hydrophobic gel by freezing or drying and offering such a solution surpassing common knowledge as enabling the konjak derivative to form a hydrophobic gel by freezing or drying.

D) Means to solve the problem

Now, the contents of this invention will be described below sequentially in the order of their occurrence.

○ Method for production of konjak derivative

In short, what is obtained by producing under insufficient conditions the conventional hydrophilic konjak derivative "which is depicted by a description that this derivative originally in a liquid or pasty state is enabled to form a hydrophobic konjak gel (not easily soluble in water) by freeze drying," namely all konjak derivatives which are not enabled to form a hydrophobic konjak by freezing and thawing constitute konjak derivatives to which this invention is applicable.

Since the scope for the production of konjak derivative contemplated by this invention is too vast to be properly covered herein, a typical example of the method will be described below. Further, since the solids content in % of konjak powder to be used is practically in the range of 1.0% - 5%, the present invention will be described below by using the range of 3 - 4% as the standard. The amount of the catalyst to be used naturally falls in the aforementioned standard range of 3 - 4%. By using 50 g of sodium citrate as a catalyst, thoroughly homogenizing the produced mixture, and keeping the stirred mixture at a temperature of not lower than 85°C

for 20 minutes, the derivative aimed at by this invention is formed. In the reaction of the same system, by using 10 g of sodium citrate and 2 g of calcium carbonate jointly as a catalyst, thoroughly homogenizing the resultant mixture, and allowing the stirred mixture to stand at a temperature in the range of 80°C - 85°C for 30 minutes, it is made possible to complete a derivative contemplated by this invention.

In the reaction in the same system described above, by selecting the relation of the amount of catalyst, the reaction temperature, and the duration of reaction properly, it is made possible to produce a derivative of this invention arbitrarily.

To arrange the foregoing description in order, in spite of the conventional theory that "reversible konjak" alone is enabled to form a hydrophobic gel by freezing, this invention has succeeded for the first time in producing a hydrophobic gel of konjak even when the konjak is not in a reversible form. This invention is based on the discovery that even the product obtained under the insufficient conditions necessary for the production of the conventional "reversible konjak," namely the conditions incapable of forming a "reversible konjak" can be embraced in this invention.

The catalysts which can be used for the derivative of this invention are caustic alkalis, alkali carbonates, alkali phosphates, organic acid salts, basic amino acids, amines, egg white, and the like. The amount of such a catalyst to be used may be in such a range as prevents the produced konjak derivative from being enabled to form a hydrophobic gel by freezing. Preferably this amount can be determined in advance by a capsule experiment.

The temperature of the reaction mentioned above is more or less varied by the kind of catalyst. It is preferred to

be determined by a capsule experiment in the range conforming to the conditions for the production of the derivative of this invention.

5 The duration of the reaction mentioned above is varied by the reaction temperature and the kind of catalyst. It is therefore preferred to be determined by a capsule experiment in the range conforming to the conditions for the production of the derivative of this invention.

10 The derivative of this invention which has been prepared as described above is enabled by the addition of a catalyst contemplated by this invention to form a hydrophobic gel through the step of freezing or heating.

15 It has been inconceivable for the derivative of this sort to form a hydrophobic gel by heating, though the prior art has enabled it to form the hydrophobic gel by freezing. This invention has realized the formation of the gel by heating owing to such discoveries as mentioned above.

○ The contents of the catalyst, namely food fibers, contemplated by this invention are as follows.

20 [Note] Literature cited: "Food fibers," compiled by Satoshi Innan and Shuhachi Kiriyama of Japan Nutritionists Society
(1) Wheat bran

25 Wheat bran, though incapable of satisfying all the conditions mentioned above, is a comparatively satisfactory source of fibers and is readily available and, therefore, is used most abundantly like powdered cellulose. The "bran" is the part formed mainly of skin which remains in the process for the production of wheat flour after the albumen and the germ have been removed. About 25% of wheat turns into the
30 "bran."

The "bran" is designated as "bran" or "shorts" by reason of size. The bran is the part formed mainly of the coarse

crust of wheat grain and the shorts is the part formed of finely divided crust mixed partly with germ and low-grade wheat. Though the composition of the bran is not fixed because it is fairly varied by the kind of wheat as raw material, the yield of flour milling, and the manner of extraction. The approximate composition is as shown in Table 1.

The "bran" tends to heighten the content of crust, increase the amount of fibers, and decrease the amount of protein in proportion as it grows in size. Neumann et al. analyzed coarse "bran" and fine "bran" to determine their compositions. According to the results of this analysis, the coarse "bran" contained 11.33% of coarse fibers, 30.5% of pentosan, and 8.74% of starch, while the fine "bran" contained 9.75% of coarse fibers, 22.5% of pentosan, and 15.65% of starch. The coarse bran and the fine bran are not distributed as classified by size but are generally shipped from the flour mill in a mixed form.

Kind and composition of domestic bran

Kind	Coarse protein	Coarse lipid	Coarse fibers	Coarse ash
General bran	14-19%	4-5%	6-11%	4.5-5.5%
*Large bran	12-16	3-5	8-13	5.0-6.0
*Small bran	13-18	4-6	6-11	4.0-5.0
*Powdered bran	15-20	4-6	5-8	3.5-4.5
Extended bran	10-17	2.5-3.5	2.5-4.5	1.5-2.8

* Rarely distributed alone, forming part of general bran

The feasibility of utilizing wheat middlings for the production of high-fiber cakes is being studied now. The wheat middlings is something like a mixture of small bran and medium bran. The content of fibers in the wheat middlings is roughly intermediate between that in the large bran and that in the

small bran.

Lorenz is now studying the feasibility of utilizing the "bran" of Triticale for bread. This rye wheat which has been grown by breeding **Duramu** wheat with rye wheat suffers from inferiority of the ability to allow production of flour, reduction in the yield of flour, and addition to the amount of "bran." The desirability of utilizing this "bran" for food has been finding recognition. The results of the analysis performed by Lorenz et al. indicate that this "bran" contains 5.5 - 6.3% of crude fibers and 18.0 - 19.3% of protein.

(2) The powdered α -cellulose has been being used heretofore for the purpose of adjusting the viscosity of food and enhancing the water-retaining property of food. It results from pulverizing the pulp derived from beeches and maple trees. The pulp is finely pulverized to such a size that not less than 90 - 95% thereof passes a mesh of 100. According to one typical set of analyses published by a certain maker, the powdered α -cellulose contains 78% of crude fibers (cf: neutral detergent having a fiber content of 99%) and 3 - 4% of pentosan and no phytic acid has a very small microorganic count of <50/g. It is marketed under the trademark designation of 'Solka Floc. Since it satisfies nearly all the conditions mentioned above, it allows very easy use and it is used nearly wholly in the production of such high-fiber food as bread.

The α -cellulose powder which is obtained by hydrolyzing pulp with an acid till it is refined and depolymerized has been introduced as a commercial product to the market.

These products of cellulose have been approved as GRAS substances by FDA.

Composition of refuse of apple remaining after squeezing (%)

	Water	Crude protein	Crude lipid	Crude fibers	Soluble nitrogen-free substance	Ash
Raw species	78.9	1.3	1.3	3.7	13.9	0.9
Dried species	10.6	4.5	5.0	15.6	62.1	2.2

(3) Beer lees

The beer lees which remains after separation by
 5 filtration of barley mash from the fermented liquor of ground
 barley in the production of beer is formed chiefly of such
 residues as the crust, bran, and germ of the hulls of barley
 and abounds in fibers. It has a very high protein content
 because it is made to add the sediment which is generated
 10 during the heating of barley mash and is formed mainly of
 protein. The composition of the beer lees (reduced to a dry
 species) based on one typical set of analyses published by
 Prentice et al. is as shown below 34.4% of protein, 3.6% of
 ash, 14.0% of crude fibers, 8.3% of crude lipid, 36.0% of
 15 acid detergent fibers, 10.0% of cellulose, and 17.0% of lignin.
 Prentice et al. are trying production of high-fiber bread
 and cookies by using the beer lees.

(4) Refuse of apple

From the amount (about 20,000 tons) of the concentrated
 20 apple juice produced in Japan today, the annual total of the
 refuse of apple resulting from squeezing apple juice is
 estimated to be about 30,000 - 40,000 tons. This refuse of
 apple is a pasty substance containing about 70% of water and
 the balance consisting mainly of fibers, insoluble pectin,
 25 and the like. One example of the composition of the refuse
 of apple is shown in Table 9.2.

The refuse of apple has been heretofore used as for

adjusting the viscosity of food. It emits nearly no odor and gives a rather sweet taste. Owing to the good image of its source which fully overcomes its relatively high cost, the utilization thereof for high-fiber food has been arresting a strong interest recently and is expected to enjoy growth in this field in the future.

(5) Bean curd refuse

The bean curd refuse is composed of 22.6% of protein, 12.3% of lipid, 44.5% of carbohydrates, 14.8% of fibers, and 6.0% of ash (reduced to dry species in accordance with the table of Japanese food compositions). Since it has high contents of fibers and protein, the utilization thereof for high-fiber food has been attracting an interest.

(6) Coconut residue

The albumen of coconut is a good source for protein. Since it has a high fiber content, however, the coconut residue which remains after extraction of coconut milk constitutes a good source for fibers. Khan et al. have been studying the utilization of coconut residue for bread and cookies. The used coconut residue has the following composition: 57.9% of carbohydrate, 16.1% of crude fibers, 18.1% of oil, 6.7% of protein, and 1.2% of ash.

(7) Other components

The defatted albumen powder of maize contains 26% of protein, 12% of fibers (acid detergent), 0.6% of lignin, etc. and can be utilized as a source for fibers. The sea grass also abounds in fibers (10.8% in kelp or 9.5% in shavings of tangle, for example) and, therefore, is useful as the source for food fibers.

Nagai et al. have been studying the utilization of the insoluble portion of burdock, holocellulose, and konjak powder for bread and Pomeranza et al. have been trying the

utilization of the hull (in a pulverized form) of oats for bread.

The food fibers which are used in this invention have been outlined.

- 5 [Note] The following symbols will be used in the following description, including working examples.

Name of food fibers	Symbol
Cellulose species	F1
Wheat bran species	F2
Apple refuse species	F3
Beer lees species	F4
Coconut residue species	F5
Maize species	F6

- 10 The catalytic effect of food fibers in this invention is conspicuous and it is specifically demonstrated by the following description.

- A konjak derivative is produced by dispersing 30 g of refined konjak powder in 1000 cc of water, adding 0.5 g of calcium hydroxide to the resultant aqueous dispersion, and
15 thorough mixing them at room temperature.

- This konjak derivative shows no noticeable change in physical properties thereof even after several days' standing at 5°C - 10°C. The konjak derivative, therefore, is caused by addition of food fibers to succumb to markedly accelerated
20 gelatin.

Example (Use of F1, an aqueous substance having a cellulose content of 2%)

(A) F1 used at a concentration of 0.028% (as solids content) for konjak derivative

- 25 (B) F1 used at a concentration of 0.006% (as solids content)

for konjak derivative

(C) F1 used at a concentration of 0.040% (as solids content)
to konjak derivative

(D) Control

- 5 When these gels are compared in surface strength by the
insertion of a needle (in terms of the duration of passage
of the needle through the gel having a fixed thickness of
15 mm), the degrees of needle insertion are as shown below.

A	B	C	D
30 minutes	20 seconds	30 minutes	4 seconds
3 mm dent	Penetration	2 mm dent	Penetration

10

It has been ascertained that the preceding incredible
gel phenomenon can be realized by the addition of food fibers.
The fact that the gelatin can be arbitrarily effected with
such a natural substance as mentioned above indicates that
15 the gel will find an unusually wide application.

The completion of the konjak derivative, namely the fact
that the konjak which cannot be converted to a gel with a
low alkali can be converted to a gel with a food fiber, is
significant for the conventional method for the production
20 of konjak.

The amount of the catalyst to be used in this invention
is such that the catalyst greatly affects the konjak derivative
even at a concentration of 0.06% as solids content.

Since the catalyst manifests a difference from the
25 control even at a concentration of 0.0001 %, this invention
finds it most favorable to determine the amount of the catalyst
to be used as occasion demands instead of particularly imposing
a lower limit on this amount.

The effect of the catalyst varies with the kind of food

fibers. This effect decreases in proportion as the size of food fibers increases. Preferably, the food fibers have a minute size and contain crude fibers in a large proportion.

While the lower limit of the amount of the catalyst to be used can be arbitrarily selected to suit the purpose of use of the formed gel, the upper limit thereof can be freely selected so long as no problem is posed concerning the taste and the palatability of the product.

The foregoing descriptions, when so tabulated as to facilitate comprehension, may be organized as shown in the following table.

(Note 1) The aqueous solution of 4% konjak powder as a solid content is used as a typical example and used in the amount of 1000 g as a standard weight.

(Note 2) The modes of embodying this invention by freezing a relevant derivative are given in the table and the modes of obtaining a relevant derivative by heating are described in working examples. The tensile strength by heating is lower on the average than that by freezing. The heating is performed at a temperature of not lower than 70°C for a period of 20 minutes as the standard.

(Note 3) Under the bracket (a) production of konjak derivative in the table, the catalyst is what is used for converting a relevant konjak powder into a corresponding derivative and the freezing is what is performed in a freezing chamber at -20°C on a sample sheet 2 mm in thickness. "No change" in the state assumed after thawing indicates the case in which the unfrozen konjak derivative of this invention exhibits the same physical properties after undergoing the steps of freezing and thawing. "Hydrophobic gel" indicates the case in which the konjak derivative of this invention is converted into a hydrophobic gel through the steps of freezing and

thawing.

The method and effect of this invention indicated in the bracket (b) of the table are the same as those explained in the bracket (a). It is provided, however, that the numerals (S1) - (S5) in the column "state after thawing" are such numerical values of tensile strength of formed hydrophobic gels as are described in detail below. The tensile strength is determined by securing a test piece 2 mm thick, 10 mm wide, and 30 mm long from a formed hydrophobic gel, hanging it vertically, fixing the upper part thereof by being nipped in a size of about 5 mm in the direction of length with a pinch cock, and allowing the hanging test piece to be left drawn downward under a prescribed load (5 g) with a pinch cock attached to the hanging terminal of the test piece in the same manner as in the upper A test piece sustaining no fracture is reported as (S1), a test piece withstanding a load up to 10 g as (S2), a test piece withstanding a load up to 20 g as (S3), a test piece withstanding a load up to 30 g as (S4), and a test piece withstanding a load up to 50 g as (S5) respectively.

(Note 4) The amount of food fibers used in this invention is indicated by the weight of solids contained therein as the standard. Where the food fibers are in the form of an aqueous 2% solution as in F1, the amount of the aqueous substance to be used is reported.

a. Production of konjak derivative				b. Method and effect of this invention			
Name of catalyst	Amount used	Time of freezing	State after thawing	Name of catalyst	Amount used	Time of freezing	State after thawing
1. Sodium citrate	Less than 35g	48 hours	No change	F1	10g	24 hours	S5
2. Sodium citrate	2g	48 hours	No change	F1	10g	24 hours	S2
3. Calcium carbonate	Less than 10g	48 hours	No change	F1	10g	24 hours	S5
4. Calcium carbonate	2g	48 hours	No change	F1	10g	24 hours	S4
5. Caustic potash	Less than 0.3g	48 hours	No change	F1	10g	24 hours	S5
6. Caustic potash	0.01g	48 hours	No change	F1	10g	24 hours	S4
7. Caustic soda	Less than 0.35g	48 hours	No change	F1	10g	24 hours	S5
8. Caustic soda	0.02g	48 hours	No change	F1	10g	24 hours	S4
9. Sodium carbonate	Less than 0.80g	48 hours	No change	F1	10g	24 hours	S5
10. Sodium carbonate	0.05g	48 hours	No change	F1	10g	24 hours	S4
11. Potassium carbonate	Less than 0.70g	48 hours	No change	F1	10g	24 hours	S5

a. Production of konjak derivative					b. Method and effect of this invention			
	Name of catalyst	Amount used	Time of freezing	State after thawing	Name of catalyst	Amount used	Time of freezing	State after thawing
12.	Potassium carbonate	0.03g	48 hours	No change	F1	10g	24 hours	S4
13.	Sodium triphosphate	Less than 3.0g	48 hours	No change	F1	10g	24 hours	S5
14.	Sodium triphosphate	0.1g	48 hours	No change	F1	10g	24 hours	S4
15.	Arginine	Less than 1g	48 hours	No change	F1	10g	24 hours	S5
16.	Arginine	0.5g	48 hours	No change	F1	g	24 hours	S2
		Less than	48 hours	No change	F1	10g	24 hours	S2

- The method of this invention is characterized in one respect by enabling the derivative of this invention not only by freezing but also by heating to form a hydrophobic gel solely through the medium of a catalyst. Specifically, the method of this invention enables the derivative of this invention by either freezing or heating to form a hydrophobic gel easily through the medium of the catalyst contemplated by this invention. Moreover, it is capable of forming ordinary konjak with a low alkali.
- The method of this invention is characterized in another respect by a veritably outstanding invention.

Specifically, when the mixed system consisting of a konjak derivative and a catalyst is in the form of an aqueous substance, what ensues is as already described above. When it is in a solid form as when the derivative and the catalyst of this invention are both solid, it means that normally the derivative is in the form of powder, flakes, film, grains, or fibers and the catalyst is in the form of powder. The mixed system is converted to a pasty state by the addition of a necessary amount of water. Thereafter, the pasty mixed system is processed by the conventional procedure to accomplish the object of this invention.

The derivative of this invention conforming to the object mentioned above is preferred to use the catalyst in a somewhat less amount within the aforementioned range of weight specified for the konjak derivative. In the case of sodium citrate of (1) - (2), for example, it is preferred to use this sodium citrate in an amount in the range of 2g- 10 g under fixed conditions.

When the aqueous derivative of this invention obtained as described above is dehydrated with a spray drier, a drum drier, or a conveyor drier into a target state to obtain a

solid derivative of this invention which has acquired improved self stability, no lower limit is particularly imposed on the ratio of the amount of the catalyst of this invention to the dried derivative as already pointed out.

- 5 ☐ The temperature used for the freezing contemplated by this invention is irrelevant so long as the conditions adopted for the freezing allow the konjak derivative of this invention to freeze.
- 10 ☐ The conditions used for the heating contemplated by this invention are irrelevant so long as they enable the konjak derivative to be heated to a temperature of not lower than 70°C.
- 15 ☐ The other substance contemplated by this invention does not need to be discriminated between the food grade and the nonfood grade but requires only to be capable of being homogeneously mixed with the konjak derivative of this invention.

20 Food grade substances: Cereals, potatoes and starches, sweeteners, cakes, oils and fats, seeds, beans, fish and shellfish, meat, eggs, milk, vegetables, fruits, mushrooms, marine plants, refreshing beverages, taste enhancers and seasonings, cooking and processing foods, synthetic pastes, and natural thickeners, binders, and stabilizers.

25 Nonfood grade substances: Typical examples of inorganic substances - Minerals, metals, ceramics, carbons, and other inorganic materials

30 Typical examples of organic substances - Synthetic resins, wood pulps, papers, cloths, fibers, organic synthetic chemicals, and medicines.

These roughly outline the substances answering the term

"other substance." More specifically, the minerals cover bentonite, kaolin, and acid clay, the ceramics cover china clay, and the synthetic resins cover ion-exchange resin and polyethylene powder. The details thereof will be described
5 in the working examples.

The amounts of these substances to be used are selected, depending on the solid-state properties of the objects aimed at and do not need to be particularly specified. They will be described in detail by way of reference in the working
10 examples.

For the sake of reference, the typical contents of the mixture of the derivative with the other substance contemplated by this invention are introduced as itemized herein below.

15 (Note 1) The mixing ratio of the derivative to the other substance according to this invention falls in the following range, (10% - 90%): (90% - 10%).

Foods

Sheets of boiled rice, sheets of potato paste, elongated
20 rods of buckwheat flour (buckwheat incapable of elongation), sheets of honey, elongated rods of bean jelly, elongated rods of corn oil, slender elongated rods of peanut, sheets of soybean paste, cylindrical masses of horse-mackerel, elongated rods of clam, sheets of pork, slender elongated rods of egg, granules
25 of milk, granules of spinach, sheets of raisin, elongated rods of dried mushrooms, macaroni-like tubes of marine plants, granules of coffee, elongated rods of mackerel shavings, slender elongated rods of mustard, sheets of curry, elongated rods of aqueous sodium alginate, elongated rods of grated
30 yam, and sheets of aqueous gelatin.

Nonfoods

Polished sheets of kaolin, polished sheets of iron oxide

red, sheets of aluminum powder, very thin sheets of ceramic material, sheets of active carbon, sheets of glass fibers, sheets of zeolite, ion-exchange membranes, fibrous masses of wood powder, paper of waterproofed surface, cloth of waterproofed surface, non-woven type cotton sheets, sheets of perfume oil, and sheets of Chinese medicine.

○ The method of forming according to this invention obtains a formed hydrophobic konjak gel or formed hydrophobic gel substance by packing the derivative of this invention or a mixture thereof with other substance in a molding frame or forming the same in the shape of granules, membranes, sheets, fibers, or noodles of freezing or heating the same during the course of forming.

In short, the final formed product can be obtained by forming the raw material in a target shape through the medium of a molding frame or freezing or heating the same during the process of forming.

○ The mixing of a food or nonfood with the derivative of this invention in accordance with this invention is only required to have the two components mixed homogeneously to an extent which suits the purpose for which the produced mixture is intended. It is, therefore, proper to select a mixing method fit for the final purpose and adjust the state of aggregate of the food or nonfood prior to the mixing.

(E) Effect of the invention

This invention has been described in detail. This invention has the following characteristic features.

① Since this invention permits its product to be distributed in a frozen state, it can extend the shelf life of the product in cold storage from the conventional average of 10 days - 20 days to not less than 6 months.

② Since the formation of a hydrophobic gel by heating which

has been unattainable heretofore is rendered feasible by this invention, it is made possible now to use the hydrophobic gel in the same way as starch and thermally coagulable protein. As a result, the utilization of the hydrophobic gel for food
5 has been markedly expanding and a method has been discovered for extensive utilization of the hydrophobic gel for nonfood as well.

These are the effects of this invention. Now, this invention will be described in detail below with reference
10 to working examples.

(F) Examples

(Note 1) As respects the konjak system used in the working examples, commercially available refined konjak powders were used as typical whole konjak systems.

15 (Note 2) Forms of catalyst used in the working examples.

F1 Aqueous substance containing 2% of solids

F2 Powdered bran

F3 Powdered refuse

F4 Aqueous 5% solution

20 F5 Powder

F6 Maize

(Note 3) Explanation of contents of Examples (1) - (38)

The term "state of 70°C" refers to the state which the konjak derivative of this invention assumes at a temperature
25 of not lower than 70°C. In the case of "paste," it is the same state as exists at normal room temperature. In the case of "gel," it is in the state of "paste" at normal room temperature and "solid" at a temperature of not lower than 70°C. Concerning a substance which retains the gel property
30 at normal room temperature, the state of "gel" is reported.

The term "after freezing and thawing" refers to the presence or absence of the formation of a hydrophobic gel

by the konjak derivative of this invention subsequently to the steps of freezing and thawing.

The term "no change" refers to the absence of a change in solid state properties before (at room temperature) and
5 after the steps of freezing and thawing, namely the fact that the relevant substance is "a paste." The term "a slight change" refers to the state of a "paste" which has started hardening. Explanation of the contents of Examples (28) - (38).

The statement that the reaction temperature and the
10 reaction time are not sufficient in spite of the use of a catalyst in the amount conforming to the conventional method portrays the formation of a hydrophobic gel by the so-called steps of heating and freezing.

"Cited Example" intended for explanation of the catalyst
15 of this invention introduces a method for the production of konjak derivatives in Examples (1) - (38). (In the working examples, the catalysts used therein were typical catalysts of the relevant systems.) The term "freezing" refers to a method for mild cooling by the use of a freezing chamber at
20 -20°C and the term "heating" refers to an operation of heating in a steamer at a temperature of not lower than 70°C.

The term "tensile strength" refers to the relative value of tensile strength of a formed hydrophobic gel. This value was determined by securing a test piece 2 mm thick, 10 mm
25 wide, and 30 mm long from a formed hydrophobic gel, hanging it vertically, fixing the upper part thereof by being nipped in a size of about 5 mm in the direction of length with a pinch cock, and allowing the hanging test piece to be left drawn downward under a prescribed load (5 g) with a pinch
30 cock attached to the hanging terminal of the test piece in the same manner as in the upper A test piece sustaining no fracture is reported as (S1), a test piece withstanding a

load up to 10 g as (S2), a test piece withstanding a load up to 20 g as (S3), a test piece withstanding a load up to 30 g as (S4), and a test piece withstanding a load up to 50 g as (S5) respectively.

5 (Note 4) Explanation of the contents of Examples (64) - (88).

These portray experiments which were performed concerning food. In the "Cited Example," (a) indicates a cited example concerning a konjak derivative and (b) a cited example concerning the use of a catalyst of this invention. The term
10 "tensile strength" refers to the same magnitude as indicated above with reference to Examples (39) - (63). The term "freezing, thawing, or heating" refers to the case of using either of the actions indicated or the case of using both of them. Example 105 covers a method for the production of
15 konjak.

(Note 5) Explanation of the contents of Examples (89) - (104). The items other than "added nonfood" are the same as those described above with reference to Examples (64) - (88).

(Note 6) Examples (1), (2), (3), (4), (39), (40), (41), (64),
20 (65), and (105) are experiments described in sentences and Examples (5) - (38), (42) - (63), and (66) - (104) are similar operations and, therefore, are reported in tables.

(Note 7) The foods and the nonfoods used in the working examples are typical examples of the relevant species.

25 Example (1)

A konjak derivative was obtained by mixing 40 g of konjak powder and 1000 cc of water, adding 34 g of sodium citrate to the resultant mixture, homogeneously mixing them together, treating the produced blend at 80°C - 87°C for 90 minutes,
30 and allowing the treated mixture to cool to room temperature.

State at 70°C	Pasty state
---------------	-------------

After freezing and thawing	No change
----------------------------	-----------

Example (2)

A konjak derivative was obtained by mixing 40 g of konjak powder and 1000 cc of water, adding 2 g of sodium citrate to the resultant mixture, homogeneously mixing them together, treating the produced blend at 80°C - 87°C for 90 minutes, and allowing the treated mixture to cool to room temperature.

State at 70°C Pasty state

After freezing and thawing No change

Example (3)

A konjak derivative was obtained by mixing 30 g of konjak powder and 1000 cc of water, adding 0.5 g of calcium hydroxide to the resultant mixture, homogeneously mixing them together, and allowing the produced blend to stand at 5°C - 15°C for 24 hours.

cool to room temperature.

State at 70°C Hard gel
(soft gel at normal temperature)

After freezing and thawing Slight change
(slightly hard paste)

Example (4)

A konjak derivative was obtained by mixing 30 g of konjak powder and 1000 cc of water, adding 0.4 g of calcium hydroxide to the resultant mixture, homogeneously mixing them together, and allowing the produced blend to stand at 5°C - 15°C for 24 hours.

cool to room temperature.

State at 70°C Hard gel
(soft gel at normal temperature)

After freezing and thawing Slight change
(slightly hard paste)

Example	5	6	7	8	9	10	11	12	13	14	15
Amount of catalyst	5g of Sodium citrate of 1g Calcium carbonate	6g of Sodium citrate of 1g Calcium carbonate	5g of Sodium citrate	6g of Sodium citrate	2g of Sodium citrate	2g of Calcium carbonate	9g of Calcium carbonate	0.05g of Sodium carbonate	0.70g of Sodium carbonate	0.03g of Potassium carbonate	0.60g of Potassium carbonate
State at 70°C	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste
After freezing and thawing	No change	No change	No change	No change	No change	No change	No change	No change	No change	No change	No change

Example	16	17	18	19	20	21	22	23	24	25	26	27
Amount of catalyst	0.02g of Caustic soda	0.33g of Caustic soda	0.01g of Caustic soda	0.28g of Caustic soda	1.0g of Sodium triphosphate	2.5g of Sodium triphosphate	0.3g of tribasic potassium phosphate	1.7g of tribasic potassium phosphate	0.5g of Arginine	1.0g of Arginine	3g of dibasic potassium phosphate	25g of dibasic potassium phosphate
State at 70°C	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste
After freezing and thawing	No change	No change	No change	No change	No change	No change	No change	No change	No change	No change	No change	No change

Example	28	29	30	31	32	33	34	35	36	37	38
Amount of catalyst	50g of Sodium citrate	10g of Sodium citrate 20g of Calcium carbonate	0.2g of Caustic soda 2g of Calcium carbonate	0.3g of Caustic soda	10g of Sodium citrate 1g of Calcium carbonate	100g of Albumen	10g of Meth potassium phosphate	0.3g of Sodium carbonate	0.5g of Caustic soda	0.5g of Sodium carbonate	10g of Sodium citrate 1g of Magnesium Carbonate
State at 70°C	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste
After freezing and thawing	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste	Paste
Reaction temperature	-80°C~	-80°C~	-80°C~	-80°C~	-80°C~	-80°C~	-80°C~	-80°C~	-80°C~	-80°C~	-80°C~
Reaction time	30 minutes	30 minutes	20 minutes	30 minutes	60 minutes	30 minutes	20 minutes	30 minutes	10 minutes	20 minutes	30 minutes

Example (39)

A powder was obtained by adding 1000 g of the konjak derivative obtained in Example (7) and 1000 cc of water together, homogeneously dispersing them, and subjecting the resultant dispersion to the action of a spray drier. A mixture was obtained by mixing about 40 g of the powder (having a water content of 10%) and 10 g of (F6).

Forty (40) g of the mixture and 900 cc of water added thereto were stirred together to swell. The swelled mass was molded into a sheet 2 mm in thickness. This sheet was kept frozen for 48 hours and then thawed to obtain a hydrophobic gel sheet. Separately, the swelled oily mass was molded into a sheet 2 mm in thickness and this sheet was kept at a temperature of not lower than 70°C for 20 minutes to obtain a hydrophobic gel sheet.

Tensile strength

(Freezing and thawing) S4

(Heating) S4

(Example 40)

A mixture was obtained by adding 1000 g of the konjak derivative obtained in Example (17) and 20g of (F1) together and thoroughly mixing them. The mixture was molded into a sheet 2 mm in thickness. This sheet was kept frozen for 48 hours and then thawed to obtain a hydrophobic gel sheet. This sheet was kept at a temperature of not lower than 70°C for 20 minutes to obtain a hydrophobic gel sheet.

Tensile strength

(Freezing and thawing) S5

(Heating) S4

Example (41)

A plate (A) was obtained by mixing 100 g of the konjak derivative obtained in Example (3) and 1.4 g of (F1), molding

the resultant mixture into a plate 15 mm in thickness, and allowing this plate to stand at normal room temperature.

A plate (B) was obtained by mixing 100 g of the konjak derivative obtained in Example (3) and 0.3 g of (F1), molding
5 the resultant mixture into a plate 15 mm in thickness, and allowing this plate to stand at normal room temperature.

A plate (C) was obtained by mixing 100 g of the konjak derivative obtained in Example (3) and 2 g of (F1), molding
10 the resultant mixture into a plate 15 mm in thickness, and allowing this plate to stand at normal room temperature.

A plate (D) was obtained by molding 100 g of the konjak derivative obtained in Example (3) in the form of a plate and allowing this plate to stand at room temperature.

(Note) Effect of repose of a varying konjak derivative after
15 2 hours' standing at normal temperature of 25°C-28°C

(Note) Effect of repose

A **shauto** weighing 10 g (K1) or 20 g (K2) on a circular bottom surface area of 7 square meters was placed on the surface
of a given konjak derivative and the time required for the
20 **shauto** to penetrate the konjak derivative was measured.

	A	B	C	D
K1	Dent of 3 mm after 30 minutes	20 seconds	Dent of 2 mm after 30 minutes	(Control) 4 seconds
K2	3 seconds	2 seconds	4 seconds	0.3 second

Example	42	43	44	45	46	47	48	49	50	51	52
Cited example	1	2	3	4	5	6	7	8	9	10	11
Amount of konjak derivative used	1000g	1000g	1000g	1000g	1000g	1000g	1000g	1000g	1000g	1000g	1000g
Form during use	Paste	Paste	Paste	Paste	Solid state	Solid state	Paste	Paste	Paste	Solid state	Paste
Konjak derivative used in solid state	Amount used in solid state				40g of powder	40g of powder				40g of powder	
	Catalyst and amount used				P5 10g	F6 5g				F2 20g	
	Amount of water added during use				900cc	900cc				900cc	
Konjak derivative used in paste state	F1 10g	F1 15g	F1 25g	F1 0.3g			F2 10g	F2 10g	F2 20g		F3 20g
	Catalyst and amount used										
Molded form	Sheet, 2mm thickness	Sheet, 2mm thickness	Film, 0.4mm thickness	Film, 0.4mm thickness	Film, 0.4mm thickness	Film, 0.6mm thickness	Granules, 2mm diameter	Granules, 5mm diameter	Plate, 7mm thickness	Elongated rods, 2mm diameter	Plate, 7mm thickness
Freezing	Freezing time	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours
	Tensile strength	S5	S2	S4	S4	S4	S3	S3	S3	S4	S5
Heating	Time of heating at 70°C	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes
	Tensile strength	S4	S2	S3	S4	S4	S3	S3	S2	S2	S3

Example		53	54	55	56	57	58	59	60	61	62	63
Cited example		12	13	14	15	16	17	18	19	20	21	22
Amount of konjak derivative used		1000g	1000g	1000g	1000g	1000g	1000g	1000g	1000g	1000g	1000g	1000g
Form during use		Solid state	Paste	Paste	Paste	Paste	Paste	Solid state	Paste	Solid state	Paste	Paste
Konjak derivative and solid state	Catalyst and amount used	F3 15g						F6 10g		F3 3g		
	Amount of water added during use							900cc		900cc		
Konjak derivative used in pasty state	Amount used	1000g	1000g	1000g	1000g	1000g	1000g		1000g		1000g	1000g
	Catalyst and amount used		P4 10g	F4 5g	F4 5g	F5 5g	F1 20g		F1 1g		F3 5g	F1 5g
Molded form		Rod, 10mm diameter	Plate, 5mm thickness	Sheet, 1mm thickness	Sheet, 1mm thickness	Hemisphere, 50mm diameter	Hemisphere, 50mm diameter	Sheet, 1mm thickness	Sphere, 20mm diameter	Sheet, 1mm thickness	Elongated rods, 2mm diameter	Elongated rods, 1mm diameter
Freezing	Freezing time	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours
	Tensile strength	S4	S5	S4	S5	S4	S5	S4	S5	S4	S4	S3
Heating	Time of heating at 70°C	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes	20 minutes
	Tensile strength	S3	S4	S3	S3	S3	S4	S4	S5	S3	S4	S2

Example (64)

A formed hydrophobic gel substance was obtained by homogeneously mixing 1000 g of the konjak derivative obtained in Example (2) and 1000 g of boiled rice, molding the resultant mixture into a sheet by following the procedure of Example (43), and freezing the molded mixture.

Tensile strength

(Freezing and thawing) S3

Example (65)

10 A formed hydrophobic gel substance was obtained by homogeneously mixing 1000 g of the konjak derivative obtained in Example (2) and 1000 g of boiled rice, molding the resultant mixture into a sheet by following the procedure of Example (43), and boiling the molded mixture in a steamer at a temperature of not lower than 80°C for 20 minutes.

15 Tensile strength

(Heating at 70°C) S2

Example	66	67	68	69	70	71	72	73	74	75	76
Cited Example	a) 3 b) 44	a) 4 b) 45	a) 5 b) 46	a) 6 b) 47	a) 2 b) 43	a) 7 b) 48	a) 8 b) 49	a) 11 b) 52	a) 3 b) 44	a) 4 b) 44	a) 22 b) 61
Amount of Konjak derivative used	1000 g	1000 g	1000 g of aqueous mass having a solids content of 4%	1000 g of aqueous mass having a solids content of 4%	1000 g	1000 g	1000 g	1000 g	1000 g	1000 g	1000 g of aqueous mass having a solids content of 4%
Added food	Potato	Buckwheat flour	Honey	Strained bean paste	Corn oil	Peanut	Soybean	Horae-macke rel	Clam	Pork	Egg
Form during use	Heat-treated paste	Powder	Honey	Paste	Liquid	Paste	Boiled paste	Raw paste (cut into fillets)	Raw paste	Mincemeat	Liquid
Amount used	500g	500g	200g	600g	300g	500g	500g	1000g	800g	1000g	500g
Molded form	Sheet	Elongated rod	Sheet	Elongated rod	Elongated rod	Slender elongated rod	Sheet	Cylindrical masses, 20mm in diameter	Elongated rod	Sheet	Slender elongated rod
Purpose of use of molded hydrophobic gel produced	Material for roll	Buckwheat (noodles)	Sheet cake	Bean jelly (elongated rod)	Solid dressing	Peanut (elongated rod)	Material for roll	Fish cake	Fish cake	Material for roll	Material for seasoning
Freezing	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	
Freezing time	S3	S3	S3	S3	S3	S3	S3	S3	S3	S3	S3
Tensile strength	20 minutes							20 minutes	20 minutes	20 minutes	20 minutes
Heating	20 minutes										
Heating time at temperature of not lower than 70°C	S2										
Tensile strength											

Example	77	78	79	80	81	82	83	84	85	86	87	88
Cited Example	a) 20 b) 59	a) 16 b) 55	a) 33 b) 45	a) 32 b) 43	a) 14 b) 53	a) 19 b) 58	a) 4 b) 39	a) 17 b) 40	a) 35 b) 59	a) 38 b) 62	a) 33 b) 63	a) 2 b) 42
Amount of Konjak derivative used	1000 g of aqueous mass having solids content of 4%	1000 g	1000 g	1000 g	1000 g of aqueous mass having solids content of 4%	1000 g	1000 g	1000 g	1000 g	1000 g	1000 g	1000 g
Added food	Milk	Spinach	Raisin	Dried mushroom	Marine plant	Coffee	Mackerel shavings	Mustard	Curry	Aqueous sodium alginate	Grated yam	Galaxy
Name of food												
Form during use	Liquid	Heat-treated paste	Granules	Raw mushroom paste	Raw marine plant paste	Fine powder	Flakes	Paste (water content 80%)	Powder	Aqueous 3% solution	Paste	Aqueous 10% paste
Amount used	500 g	1000 g	1000 g	1000 g	300 g	300 g	300 g	300 g	200 g	200 g	500 g	300 g
Molded form	Granules, 5 mm in diameter	Granules, 5 mm in diameter	Sheet	Elongated rod	Macaroni-like tubes	Granules, 3 mm in diameter	Elongated rod	Slender elongated rod	Sheet	Elongated rod	Elongated rod	Sheet
Purpose of use of molded hydrophobic gel produced	Cake	Salad	Cake or roll	Material for seasoning	Material for seasoning	Ground coffee	Material for seasoning	Material for seasoning	Material for seasoning	Material for seasoning	Noodles of grated yam	Material for seasoning
Freezing	48 hours	48 hours	48 hours			48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours
Freezing time	48 hours	48 hours	48 hours			48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours
Tensile strength	S3	S2	S4			S4	S3	S3	S3	S3	S3	S2
Heating				20 minutes	20 minutes						20 minutes	
Heating time at temperature of not lower than 70°C												
Tensile strength				S2	S2						S3	

Example	89	90	91	92	93	94	95	96	97	98
Cited Example	a) 2 b) 43	a) 3 b) 44	a) 13 b) 52	a) 4 b) 61	a) 30 b) 62	a) 35 b) 46	a) 25 b) 59	a) 19 b) 58	a) 16 b) 55	a) 2 b) 43
Amount of Konjak derivative used	1000 g	1000 g	1000 g	1000 g	1000 g	1000 g	1000 g	1000 g	1000 g	1000 g
Added nonfood	Name of food	Kaolin	Aluminum	China Clay (mixture)	Activated carbon	Grass fiber	Zeolite	Ion-exchange membranes	Wood powder	Paper
	Form during use	Powder	Powder	Powder	Powder	Fiber	Fine masses	Small granules (Sphere)	Powder	Film
	Amount used	700 g	200 g	1000 g	300 g	200 g	300 g	500 g	300 g	2 m'
Purpose of use of molded hydrophobic gel produced	Molded form	Sheet	Sheet	Sheet	Sheet	Sheet	Sheet	Sheet	Fiber	Film
	Grinder	Grinder	Electromagnetic wave	Ceramic film	Film for decolorization	Grass fiber sheet	Absorbent film	Ion-exchange membranes	Heat insulator	Waterproof paper
	Freezing time	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours	48 hours
Heating	Tensile strength	S3	S3	S3	S3	S3	S3	S3	S3	Not lower than S5 (containing paper strength)
	Heating time at temperature of not lower than 70°C		20 minutes			20 minutes			20 minutes	
	Tensile strength		S2			S3			S2	
Remarks			Used as dried	Used as dried	Used as dried	Used as dried	Used as dried	Used as dried	Used as dried	Used as dried

Example	99	100	101	102	103	104
Cited Example	a) 2 b) 43	a) 3 b) 44	a) 4 b) 45	a) 1 b) 48	a) 9 b) 48	a) 9 b) 48
Amount of Konjak derivative used	1000 g	1000 g	1000 g	1000 g	1000 g	1000 g
Added nonfood	Cloths (Cotton) Cloths	Cotton (Cotton) Cotton	Citronellal Liquid	Licorice Powder	Pursultiamine Powder	Niacinamide Powder
Form during use						
Amount used	1.5m'	600 g	100 g	300 g	30 g	30 g
Molded form	Sheet	Non-woven type cotton sheet	Sheet	Sheet	Small granules	Small granules
Purpose of use of molded hydrophobic gel produced	Sheet	Non-woven type cotton cloths	Sheet of perfume oil	Sheet for extraction (for medical use)	Capsule (for medical use)	Capsule (for medical use)
Freezing	Freezing time	48 hours	48 hours	48 hours	48 hours	48 hours
	Tensile strength	Not lower than S5 (containing cloth strength)	S3	S4	S3	S3
Heating	Heating time at temperature of not lower than 70°C					
	Tensile strength					
Remarks	Used as dried	Used as dried		Used as dried	Used as dried	Used as dried

Example (105)

A cake of konjak was produced by dispersing 30 g of konjak power in 1000 cc of water, causing the dispersion to swell, mixing the swelled mass with 0.5 g of calcium hydroxide and
5 10 g of F1, throughly mixing them, placing the mixed mass in an angular frame, and subjecting it to a heat treatment at a temperature of not lower than 70°C for 30 minutes.

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FROM: Carole Seaton for Diane Dunn McKay **NO. OF PAGES (with cover):** 38

RE: Our Ref. No. 4296-105.1 US

DATE: November 25, 2003

MESSAGE

Dear Examiner Tran:

Please find the attached as per our conversation. I will obtain a copy of the first page of the English translation and forward same to you via facsimile. Thank you for your attention to this matter. Please do not hesitate to contact me should you have any questions.

Very truly yours,

Carole Seaton